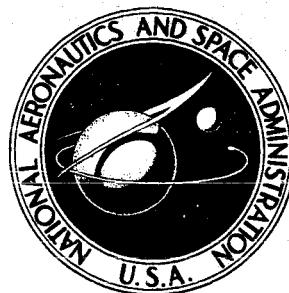


**NASA TECHNICAL
MEMORANDUM**



NASA TM X-2964

NASA TM X-2964

**TENSILE PROPERTIES FROM
ROOM TEMPERATURE TO 1315° C
OF TUNGSTEN-LINED TANTALUM-ALLOY
(T-111) TUBING FABRICATED BY
HOT ISOSTATIC PRESSING**

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION • WASHINGTON, D. C. • FEBRUARY 1974

1. Report No. NASA TM X-2964	2. Government Accession No.	3. Recipient's Catalog No.	
4. Title and Subtitle TENSILE PROPERTIES FROM ROOM TEMPERATURE TO 1315⁰ C OF TUNGSTEN-LINED TANTALUM-ALLOY (T-111) TUBING FABRICATED BY HOT ISOSTATIC PRESSING		5. Report Date February 1974	
		6. Performing Organization Code	
7. Author(s) Robert J. Buzzard and Robert R. Metroka		8. Performing Organization Report No. E-7628	
9. Performing Organization Name and Address Lewis Research Center National Aeronautics and Space Administration Cleveland, Ohio 44135		10. Work Unit No. 503-25	
		11. Contract or Grant No.	
12. Sponsoring Agency Name and Address National Aeronautics and Space Administration Washington, D.C. 20546		13. Type of Report and Period Covered Technical Memorandum	
		14. Sponsoring Agency Code	
15. Supplementary Notes			
16. Abstract <p>This study involved determining the effects of a thin tungsten liner on the tensile properties of T-111 tubing considered for fuel cladding in a space power nuclear reactor concept. The results indicate that the metallurgically bonded liner had no appreciable effects on the properties of the T-111 tubing. A hot isostatic pressing method used to apply the liners is also described. And a means for overcoming the possible embrittling effects of hydrogen contamination is presented.</p>			
17. Key Words (Suggested by Author(s)) Metals Materials Tantalum alloy Nuclear reactor materials		18. Distribution Statement Unclassified - unlimited	
19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 20	22. Price* \$2.75

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SUMMARY

The tensile properties of tungsten-lined T-111 (Ta-8W-2Hf) tubing, as determined from room temperature to 1315° C, were compared with those of unlined T-111 tubing in this study. The results indicate no appreciable effect of the metallurgically bonded liner on the properties of the tubing. The properties of both lined and unlined T-111 were essentially the same at all test temperatures, except for a lower total elongation of the lined material at test temperatures of 1200° C and higher. At low test temperatures (425° C and lower), the tungsten cracked during testing but remained bonded to the T-111. At higher test temperatures (650° C and higher), the tungsten deformed uniformly with the T-111.

The tungsten-lined tubing was fabricated by a hot isostatic pressing technique. Tungsten foil was wrapped around a hollow mandrel to the desired total liner thickness. The wrapped mandrel was placed inside the T-111 tubing and hot isostatically pressed in an autoclave. This resulted in solid-state welding of the tungsten layers to each other and to the inside wall of the T-111 tubing. Excellent dimensional control was achieved, and the resulting liners were smooth and wrinkle free.

The pressed material was brittle when flattened diametrically at room temperature, presumably as a result of hydrogen introduced into the T-111 from the autoclave atmosphere. But a low-temperature vacuum anneal (1 hr at 425° C) decreased the hydrogen content to less than 2.5 ppm and restored ductility to the T-111.

INTRODUCTION

A compact, liquid-metal-cooled, fast-spectrum, space power reactor concept was

studied at the NASA Lewis Research Center (ref. 1). The reactor operating goals were 50 000 hours at a fuel-pin cladding temperature of 980° C.

In this concept, the tantalum-base alloy T-111 (Ta-8W-2Hf) was selected for intensive study as the fuel-pin cladding material, with uranium mononitride (UN) as the fuel and lithium as the coolant (ref. 2). A thin, 0.13-millimeter (0.005-in.), tungsten liner was to be used to separate the fuel from the cladding and thereby prevent chemical reaction between the UN and the T-111. The tungsten liner initially does not have to be metallurgically bonded to the T-111. However, it is expected that bonding will occur during reactor operation because of stresses imposed upon the liner as a result of irradiation-induced swelling of the UN. Therefore, this study was conducted primarily to determine whether this bonded liner would affect the strength and ductility of the T-111 and thereby alter its capability to perform its designed functions. Tungsten foil was metallurgically bonded to the inner surface of T-111 tubes and the resultant composite structure was tensile tested. The tensile test results were then compared with those for unlined T-111. Several specimens were also annealed after pressing to obtain a greater diffusion zone between the tungsten and T-111 than that expected at the 980° C cladding temperature. This greater diffusion zone should represent the maximum expected after reactor operation for several thousand hours at a fuel-pin cladding temperature of 1270° C. This higher cladding temperature could result from increasing the reactor operating temperature should greater reactor efficiency be desired in this system in the future.

Prior to this study, the principal method used to apply tungsten liners to T-111 tubing was the thermal expansion method described in reference 3. However, this method does not produce complete metallurgical bonding of the liner to the cladding. So another fabrication method was needed to produce the bonding required to simulate the longer term operational mode of this cladding system. Thus, a secondary purpose of the study involved development of a hot isostatic pressing (HIP) method for solid-state welding of tungsten foil to the inner surface of T-111 tubing.

The results of the bonding study and the subsequent evaluation tests are summarized in this report. The evaluation included tensile tests (from room temperature to 1315° C), room-temperature tubing crush tests, microstructural examinations, and chemical analysis.

EXPERIMENTAL METHODS

Material

The T-111 (Ta-8W-2Hf) tubing used in this study was commercially produced to an

outside diameter of 19 millimeters (0.75 in.) and a wall thickness of 1.5 millimeters (0.060 in.). The tubing was given a 1-hour recrystallization-anneal at 1650^o C, followed by a 1-hour anneal at 1315^o C. The latter anneal represents a postweld stress relief anneal that would be given to actual fabricated reactor hardware. The chemical analysis of this material is shown in table I.

The tungsten foil used as the liner material was commercially produced by powder metallurgy methods. It was received in the wrought condition as 0.025-millimeter (0.001-in.) thick foil. The chemical analysis of this material is also shown in table I.

Lining Procedure

The tungsten-lined T-111 tubing used as test specimen stock in this study was fabricated by a hot isostatic pressing process performed in a high-temperature, high-pressure autoclave. The tooling assembly for this process is shown schematically in figure 1. A hollow-internal-mandrel technique was used. In this technique, pressure is applied to the inside surface of the internal mandrel to force the tungsten against the inner wall of the T-111 tubing. Hollow mandrels were used in an effort to prevent wrinkling of the liner. Wrinkling was observed in reference 4, where a solid-mandrel technique was tried. With the hollow-mandrel technique, a solid-state weld was achieved between the tungsten and the T-111, as well as between the layers of tungsten foil which comprised the liner.

The procedure used in this technique is described in the following paragraphs.

Preparation for hot isostatic pressing. - The T-111, the molybdenum tubing, and the tungsten foil were cut to the lengths indicated in figure 1. The 0.025-millimeter (0.001-in.) thick tungsten foil was cut to a width of 248 millimeters (9.75 in.) so that it could be wrapped five times around the internal molybdenum mandrel with no overlap of material occurring at the seam. The molybdenum mandrels extended beyond the tungsten and T-111 tubing at each end to allow for insertion of end seals made from short pieces of molybdenum tubing. The wall thickness of these end seals was equal to the combined tungsten and T-111 thicknesses and the assembly gap distance.

The molybdenum mandrels were machined to the nominal dimensions shown in figure 1. The wall thickness of the inner mandrel was designed to allow it to expand outward during the HIP process. The outer mandrel was of sufficient thickness to resist outward expansion of the T-111 tubing. Assembly gaps, as shown in the figure, were allowed between mating components to permit easy assembly of the parts.

The outside diameter of the T-111 tubing was checked to ensure that all T-111 pieces used in this study were within ± 0.012 millimeter (± 0.0005 in.) of the nominal 19.1-millimeter (0.75-in.) diameter required.

All the component parts were sanded with 600-grit paper to remove any surface oxides, burrs, and so forth, from the material. All material was first scrubbed then ultrasonically cleaned in solutions of detergent and hot water. This process was followed by separate rinses in acetone, methyl alcohol, and distilled water in the ultrasonic cleaner. After this treatment, all material was handled with clean, lint-free white gloves. The materials were further cleaned and degassed by heating for 1 hour at about 1100°C . The molybdenum was heated in a hydrogen atmosphere, while the tungsten and T-111 were heated in a 10^{-5}-N/m^2 ($\sim 10^{-7}$ -torr) vacuum. For this treatment, the tungsten was loosely formed into its wrapped cylindrical configuration in order to fit it into the furnace and to simplify the wrapping procedure during final assembly of the components.

After furnace cleaning, all components were assembled preparatory to welding. The assembled components were again furnace cleaned in vacuum. Immediately upon removal from the furnace, they were sealed in plastic bags containing a high-purity inert-gas atmosphere and transferred to an electron-beam-welding chamber. The chamber was evacuated to about 10^{-3} N/m^2 (10^{-5} torr), and the ends of the capsules were sealed by electron beam welding. The capsules were leak checked by pressurizing them in helium and immediately immersing them in methyl alcohol to observe bubbles.

After ensuring that the capsules were leak free, we sent them to a contractor's facility for hot isostatic pressing. Twenty-eight such capsules were prepared in this manner.

Hot isostatic pressing. - The capsules were again leak checked at the contractor's facility prior to the hot isostatic pressing operation. They were then individually wrapped in tantalum and molybdenum foil. The entire group which comprised a pressing run (i.e., 10 capsules, maximum) was similarly wrapped to absorb (getter) any residual impurities in the autoclave during the pressing run. The specimen load was then placed in the autoclave heat zone. The chamber was sealed, flushed twice with helium, and heated slowly (for about 3 hr) to the pressing temperature as helium pressure was gradually applied. The temperature was held at 1650°C for about 5 minutes at a gas pressure of 206.8 MN/m^2 (30 ksi). The temperature gradient along the specimen load was about $\pm 8^{\circ}\text{C}$, as indicated by tungsten - 5-percent-rhenium/tungsten - 26-percent-rhenium thermocouples placed at three locations along the load. At the end of the cycle, the heater was turned off and the autoclave was slowly depressurized. The individual capsules were again leak checked after removal from the autoclave and then returned to Lewis for decanning.

Postpressing procedures. - The molybdenum was removed from the capsules by first cutting off the molybdenum end pieces and then leaching the mandrels in a solution of 50 parts water, 50 parts nitric acid, and 5 parts sulfuric acid. Both the external mandrel and the hollow internal mandrel dissolved in about the same length of time.

After the molybdenum was completely dissolved, the specimens were placed in a strong sodium hydroxide solution for several minutes, washed in water, and then placed in fresh leaching solution. To assure complete removal of the molybdenum, this procedure was repeated several times. Leaching time was approximately 1 hour per tube. This operation was accomplished quickly because of the small amount of molybdenum present as mandrel material.

After leaching, the tubes were rinsed in separate acetone and methyl alcohol baths. They were then vacuum annealed at about 10^{-5} N/m² (10^{-7} torr) for 1 hour at 1315° C for cleaning and to remove excess hydrogen which may have been introduced into the T-111 during the pressing operation (as discussed in the section Flattening Test Results). This anneal was omitted for tubing used in flattening tests, as these test results were to be based upon various annealing histories. Finally, micrometer measurements of the outside diameter and wall thickness were taken at several locations on each specimen to indicate whether any dimensional changes occurred as a result of the pressing operation.

A few of the tubes were surface ground on the outside diameter, using a centerless grinder, to determine whether such treatment would crack or otherwise affect the tungsten liners.

Test Equipment and Procedures

After fabrication and inspection of the tungsten-lined T-111 tubing was completed, the material was machined into specimens for use in flattening tests and tensile tests. The following procedures were used in these operations:

Specimen preparation. - Specimens intended for use in flattening tests consisted of short, 6.35 millimeter (0.25 in.), ring-shaped cylinders. These cylinders were cut from as-pressed tungsten-lined T-111 tubes with a 120-grit silicon carbide cutoff wheel. The cut ends of the rings were finished with 600-grit polishing paper. The specimens were cleaned as described previously, except that the furnace anneal was omitted.

Tensile specimens were fabricated by first slitting 76-millimeter (3-in.) long sections of tungsten-lined T-111 tubing lengthwise into equal thirds, as indicated in figure 2, and then grinding to the configuration shown. The grinding direction was from the tungsten toward the T-111. This eliminated the possibility of chipping the tungsten liner. The grip holes were electrodischarge machined (EDM). The longitudinal seam in the tungsten liner was not included in the test section of any specimen. The ground edges of the gage section were sanded longitudinally with 320-grit grinding paper. The specimens were cleaned as previously described. They then were annealed for 1 hour at 1315° C in vacuum for cleaning and to ensure stress relief of the machined and sanded

edges of the gage section. Several of the rings and tensile specimens were further vacuum annealed for 3 hours at 1650°C to obtain a diffusion zone of about 0.025 millimeter (0.001 in.) between the tungsten and the T-111.

After the annealing operations were completed, the tensile specimens were marked for identification, and pretest dimensional measurements were made. The initial gage length was determined by measuring the distance between two scribe marks located at a nominal 25-millimeter (1-in.) interval on the convex face of each specimen. The thickness and width of the gage section of each specimen were measured with a micrometer. The thickness of the lined specimens included the tungsten liner. Since the cross section of each specimen was in the form of a pie-shaped segment, the width was measured as a chord of the outside diameter of the tube. And since calculation of area based on such measurements was tedious, a nomograph was developed for this purpose.

Flattening tests. - Flattening tests were performed to evaluate the effect of hot isostatic pressing and of subsequent anneals at various temperatures on the ductility of the tungsten-lined T-111 tubing. The behavior of the tungsten liner was also evaluated in these tests. Flattening tests were performed in air at room temperature ($\sim 25^{\circ}\text{C}$). The rings were placed between the crossheads of a hydraulically operated compression testing machine and deformed diametrically at a crosshead movement rate of 25 millimeters (1 in.) per minute. The test was terminated when cracking of the T-111 occurred or when the rings were completely flattened.

Tensile tests. - Tensile testing was performed by using a screw-driven tensile testing machine. The output of the load cell of the machine was fed into a strip-chart recorder so that data were recorded as load against time, the time axis being referred to crosshead movement rate. All testing was done at a crosshead movement of 1.3 millimeters per minute (0.05 in./min).

The testing furnace consisted of a stainless-steel, O-ring-sealed vacuum chamber with a double-walled, water-cooled shell. The system was capable of attaining a vacuum of the order of $1.3 \times 10^{-4} \text{ N/m}^2$ (10^{-6} torr). However, during specimen heatup, the pressure rose to about $2.6 \times 10^{-3} \text{ N/m}^2$ (2×10^{-5} torr).

The specimens were radiantly heated by a resistance-heated tungsten split-tube heating element powered by a variable transformer. Temperature was measured with a platinum/platinum - 13-percent-rhodium thermocouple tied to the center of the specimen's gage length. The accuracy of temperature measurement was estimated to be about $\pm 3^{\circ}\text{C}$.

Special molybdenum tensile grips were fabricated for use in this program. The two-piece design shown in figure 3 was used because the curved surface, which positions the specimen properly for axial loading, could readily be ground eccentrically to a high degree of accuracy and parallelism with the load axis. Also, minor adjustments in specimen positioning due to variations in specimen thickness could be accommodated

very easily with this design. Tensile tests were performed from room temperature to 1315° C, for unlined and lined specimens annealed for 1 hour at 1315° C. Test specimens which were annealed for 3 hours at 1650° C were tensile tested at room temperature, 980° C, and 1315° C.

Post-test examination. - After tensile testing, the distance between gage marks was measured again for use in elongation calculations. The nature of the fractures was such that meaningful post-test measurements of cross-sectional areas could not be obtained for use in reduction-in-area measurements even though attempts were made both optically and with micrometers.

Part of the broken gage sections of selected tensile specimens, and several of the flattening test specimens, were submitted for microstructural observation. The T-111 etchant used in the metallographic observations was a bifluoride swab etchant, consisting of a solution of 50 cubic centimeters nitric acid, 20 cubic centimeters water, and 30 grams of ammonium bifluoride. The etchant used for the tungsten liners was Murakami's reagent (i.e., 10 g KOH, 10 g K_3FeCN_6 , 100 cm³ H₂O).

RESULTS AND DISCUSSION

Hot Isostatic Pressing Results

Postpressing inspection of the hot isostatically pressed capsules indicated that all 28 tubes had bonded successfully. No leaks were found in the capsules after pressing and after mandrel leaching, and the liners appeared smooth and intact. No delaminations or other imperfections were evident in any of the tubes.

One of the tungsten-lined T-111 tubes is shown in figure 4. A small section of the tube was cut away by using a hand-fed, water-cooled cutoff wheel as described previously. The removed section was polished with 1- to 5-micrometer (medium) diamond paste and etched to facilitate comparison with the as-cut edge. As indicated in the enlarged view, no delaminations or severe chipping as a result of the cutting operation were observed on any of the cut edges.

Also shown in figure 4 is a cross section (end view) of the tube at the seam in the liner. The thickness of the tungsten in this area varied by the thickness of one wrap of tungsten foil, because the length of the foil was not quite sufficient to allow five full wraps around the inner mandrel. This situation could be corrected by more accurate measurement of the foil length. Also, the wedge-shaped gap at the edge of the foil could be eliminated by proper shaping of the foil edge, either by machining or by chemical milling. However, we believe that neither of these situations affected the intended use of the tubing for the purposes of this study.

Measurements taken at various locations on each tube verified that the dimensions did not change appreciably as a result of the pressing operation. The greatest variation in outside-diameter measurement was about ± 0.005 millimeter (0.0002 in.), which includes measurement errors as well as possible errors in locating the exact measurement points on which original dimensions were based.

Additional centerless grinding of the outside diameter of several of the lined tubes was performed successfully, with no adverse effect on the tungsten liner. This information was obtained should the need for such operations ever arise during applications of this material beyond the needs of this study.

Flattening Test Results

Preliminary flattening tests indicated severe embrittlement of the as-pressed material. Such embrittlement had been observed previously for T-111 sheet which had been hot isostatically pressed in the same autoclave as was the material of the present study. This embrittlement was attributed to hydrogen contamination during the pressing operation. This contamination is assumed to be caused by decomposition of small amounts of residual moisture in the autoclave. Chemical analysis of the tubes from this present study indicated hydrogen pickup during bonding to levels of about 25 ppm (table I).

Vacuum anneals of 1 hour at 425°C were sufficient to reduce the hydrogen content to less than 2.5 ppm and to restore ductility as measured by the flattening tests. The material is expected to receive a vacuum anneal of 1 hour at 1315°C as an application-oriented postweld stress-relief treatment. Multiple short-term anneals of this type have been shown in other studies (unreported work at Lewis) not to affect the properties of the material. Therefore, this anneal was repeated for the tensile specimens of this study to ensure complete removal of all hydrogen from the material. A photomicrograph of a tubular specimen which was vacuum annealed for 1 hour at 1315°C and subsequently flattened upon itself at room temperature is shown in figure 5(a). The tungsten liner cracked, as would be expected, but remained attached to the ductile T-111 even after severe deformation. The chemical analysis for this specimen indicated a hydrogen level of less than 0.5 ppm (table I).

The results of a flattening test performed on a tungsten-lined T-111 ring annealed for 3 hours at 1650°C were similar to those of the rings annealed for 1 hour at 1315°C ; that is, the ring was completely flattened with no cracking observed in the T-111. A photomicrograph of this ring is also shown in figure 5(b).

Tensile Test Results

The results of tensile tests for both unlined and tungsten-lined T-111 tubing specimens are shown in figure 6. A secondary peak in strength is evident at test temperatures of about 800° to 900° C for both materials. This strength peak has been observed previously (refs. 5 and 6) and is believed to be the result of a dynamic strain-aging effect which is sensitive to the amount of residual oxygen in solid solution in the T-111. Since the observed variation in peak strength between the lined and unlined tubing in this temperature range was only about 5 percent, it is reasonable to assume that the mechanics of this strain-aging effect were not influenced greatly (if at all) by adding the tungsten liner to the T-111 material or by the thickness of the tungsten - T-111 diffusion zone.

The yield strength of both the lined and unlined T-111 agreed very well at all test temperatures (fig. 6). Yield strength decreased continuously with increasing test temperature. The drop-in-yield phenomenon which was observed previously for sheet-type T-111 (ref. 6) was absent from the load-strain curves obtained in the present study. However, some flattening of the curves immediately past the yield point was common to both tubing materials at test temperatures of 760° C and lower. Serrations in the load-strain curves were observed in both materials at strains beyond the yield point, at test temperatures of about 760° to 980° C.

The presence of the tungsten liner was apparent in the load-strain plots of one of the two room-temperature tensile tests and the 1315° C test. A slight drop in load was observed in the strain-hardening portion of the curves, which was rationalized as a local separation of the tungsten.

Total elongation of T-111 was not affected by the tungsten liner at test temperatures of about 1000° C and lower. At higher test temperatures, total elongation of lined T-111 tubing remained at about 20 percent, while that of the unlined tubing increased to about 40 percent. The higher test temperatures are in a region in which the deformation mode of T-111 changes from intragranular fracture to intergranular separation resulting from grain boundary sliding. Hence, total elongation becomes a measure of grain boundary voidage, or gaps, rather than a true indication of ductility. Perhaps a better indication of ductility, or deformation characteristics, can be had by observing the specimens shown in figure 7 and the photomicrographs of figure 8.

Figure 7 shows the tungsten-lined surface of several of the specimens tensile tested in this study. The change in surface texture with increasing test temperature can be seen in this figure. At low test temperatures (425° C and lower), the tungsten cracked but remained attached to the T-111. At higher test temperatures (650° C and higher), the tungsten deformed smoothly without severe surface cracking. (The apparent spalling

of tungsten on the room-temperature test specimen was the result of attempts to remove the tungsten with a pair of pliers after tensile testing.)

The photomicrographs of figure 8 represent the type of fractures observed in this study. At test temperatures of 25° and 315° C, figures 8(a) and (b), the tungsten liner cracked in a brittle manner and separated, and the more ductile T-111 strained locally beneath the separated areas.

At the higher test temperatures, the increased ductility of the tungsten allowed it to deform along with the T-111. Figure 8(d) illustrates that the lined material deformed as a single entity, indicating that the liner was still bonded to the T-111. The fractured ends of the specimens indicate a high degree of ductility in the T-111 at test temperatures as high as 1200° C (fig. 8(e)). At this test temperature, grain boundary sliding has begun to play an important role in the fracture mechanism, and the reduction in area observed for the specimens as a whole appears to have decreased because of individual grain boundary separations along the plane of fracture.

APPLICABILITY TO REACTOR USE

The HIP method used herein to fabricate tungsten-lined T-111 tubing and the resulting properties of the lined material should be directly applicable to the space power nuclear reactor concept described in reference 1. This fabrication method produced a consistent, high-quality lined tubing whose properties were similar to those of unlined T-111 tubing except for the brittleness of the tungsten at low temperatures. Although this method requires the machining to proper dimensions of the two molybdenum tubing mandrels plus two end plugs, the machining tolerances are no more critical than those which can be obtained using standard shop practices and therefore should not present a problem in machining when applied to reactor needs. More importantly, the assembly gaps provided easy insertion of the tungsten-wrapped liner mandrel into the bore of the T-111 tubing. These assembly gaps greatly eased the assembly of the tubes as compared with other methods which require very tight fits. As an example, each of the tubes used in this study was assembled and ready for welding in about 1 minute as compared with assembly times as long as several hours with other fabrication methods. This is an important factor for consideration when applying liners to longer tubes such as those required for reactor fuel-pin cladding. In pressing longer tubes by this method, longer autoclave heat zones are required than were used in this study. However, facilities are available with uniform heat zones about 500 millimeters long. Thus, full-length (430-mm) fuel elements for use in the NASA Advanced Power Reactor Concept could be lined with this process.

Before applying this fabrication method to the reactor in its present stage of de-

velopment, several other areas should be investigated. For example, additional evaluations based on variations in the thickness of the tungsten - T-111 diffusion zone might be desired. Although present reactor concepts do not require a weld between the liner and the T-111, some degree of welding, or a slight diffusion zone, was desirable in this study to obtain test specimens whose tungsten - T-111 interface would be representative of that anticipated at a time beyond the initial stage of normal reactor operation. Should a study of various thicknesses of diffusion zones be desired, to represent the condition of the cladding under a variety of fuel-pin operating conditions, postbonding anneals could be used to serve this purpose.

Modifications to the tungsten liner should also be made to improve the seam at the point where the ends of the wrap meet. For example, no overlap of tungsten occurred in the tubes pressed in this study, but a slight gap did occur in which the liner was only 0.102 millimeter (0.004 in.) thick (fig. 4). This gap could be eliminated by more exact measurement of the length of the tungsten foil used as the liner wrapping. In addition, beveling or chemical milling of the ends of the foil would provide a smoother joint at the seam, should this be desired.

CONCLUDING REMARKS

The property measurements performed in this study indicated that a thin, bonded tungsten liner had no significant effect on the properties of T-111 tubing. The tensile properties of the lined material were similar to those of the unlined T-111. Also, separation of the tungsten liner during the lower temperature tensile tests did not appear to act as a significant stress-raiser for the underlying T-111.

Further investigation of this nature would be desirable, however, using tungsten-lined T-111 whose tungsten - T-111 interface has experienced a greater range of inter-diffusion than that represented in this study. For example, preliminary calculations have indicated that the diffusion zone after 50 000 hours might be of the order of 1.5 micrometers (6×10^{-5} in.) at 1000° C or 73 micrometers (0.003 in.) at 1270° C. And most importantly, the creep characteristics of the material should be investigated from a metallurgical viewpoint and to obtain design-type data. Such investigation should include hoop-type loading, as well as uniaxial-type loading, to more closely simulate the type of loading that will be experienced under actual operational conditions.

SUMMARY OF RESULTS

This study was primarily concerned with determining the effects of a thin, bonded

tungsten liner on the tensile properties of T-111 tubing considered for fuel cladding in an advanced space power nuclear reactor concept. The following conclusions were drawn from this study:

1. A thin (12.7 mm) tungsten liner metallurgically bonded to the inner surface of 152-millimeter-thick T-111 tubing has no appreciable effect on the tensile properties of the tubing, nor does the presence of a 0.025-millimeter-thick tungsten - T-111 diffusion zone.

2. Hot isostatic pressing offers good potential for use in applying metallurgically bonded tungsten liners to T-111 tubes used for reactor claddings.

3. The embrittling effects of possible hydrogen contamination during hot isostatic pressing can be eliminated by subsequent vacuum anneals at 425⁰ C or higher.

Lewis Research Center,

National Aeronautics and Space Administration,

Cleveland, Ohio, September 27, 1973,

503-25.

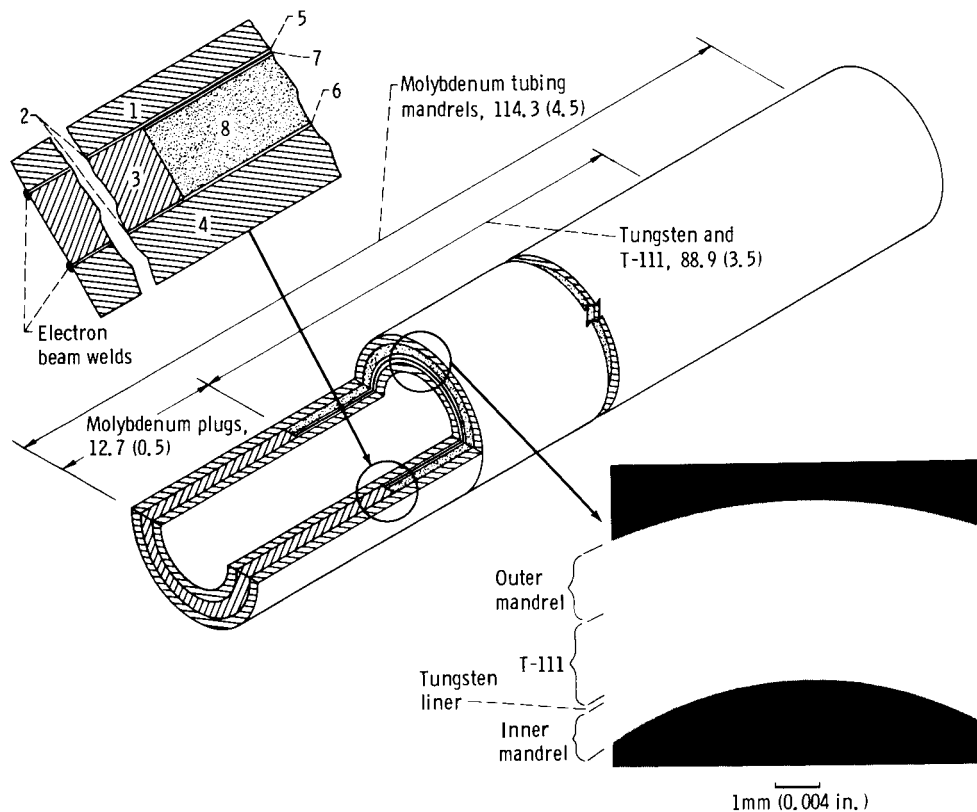
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TABLE I. - TYPICAL CHEMICAL ANALYSIS OF T-111 TUBING AND TUNGSTEN-
LINER MATERIAL USED IN THIS STUDY

Material	Major alloying elements, ^a wt. %			Other elements, ^a ppm by weight				
	Ta	W	Hf	C	O	N	H	Zr
T-111 tubing	Balance	7.9	2.3	48	33	30	3.2	440
Tungsten foil	-----	Balance	---	30	43	<10	5	---
Tungsten-lined T-111 tubing:								
As-pressed	-----	-----	---	--	--	---	25	---
Pressed and vacuum annealed at 425 ^o C for 1 hr	-----	-----	---	--	--	---	<2.5	---
Pressed and vacuum annealed at 1315 ^o C for 1 hr	-----	-----	---	--	35	8	<.5	---

^aCarbon content determined by combustion. Oxygen, nitrogen, and hydrogen determined by vacuum fusion. Remaining elements determined by wet chemistry.



Component	Inside diameter		Outside diameter		Nominal thickness	
	mm	in.	mm	in.	mm	in.
1 - Inner mandrel	14.07	0.554	15.60	0.614	0.77	0.030
2 - Plug assembly gaps	---	---	---	---	.025	.001
3 - Molybdenum plug	15.67	.617	19.08	.751	1.70	.067
4 - Outer mandrel	19.13	.753	21.70	.853	1.29	.050
5 - Tungsten - T-111 assembly gap	---	---	---	---	.08	.003
6 - T-111 - outer mandrel assembly gap	---	---	---	---	.05	.002
7 - Five tungsten wraps	---	---	---	---	.127	.005
8 - T-111 tubing	16.00	.630	19.05	.750	1.53	.060

Figure 1. - Configuration of T-111 tubing, tungsten liner, and molybdenum mandrels used in hot isostatic pressing procedure. (Dimensions are in mm (in.)).

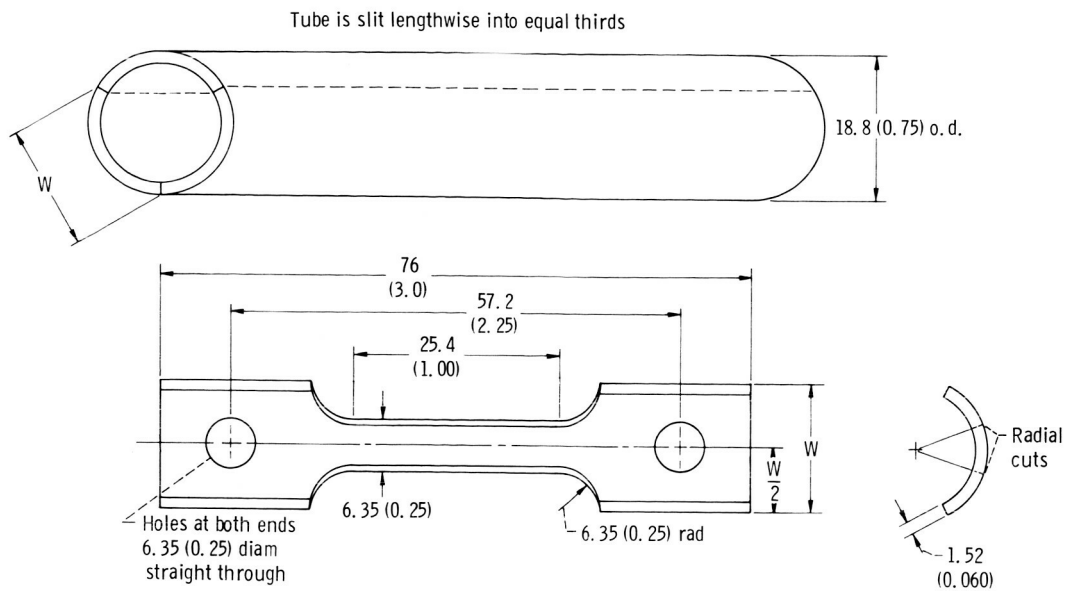


Figure 2. - Tubing-section tensile test specimen. (Dimensions are in mm (in.).)

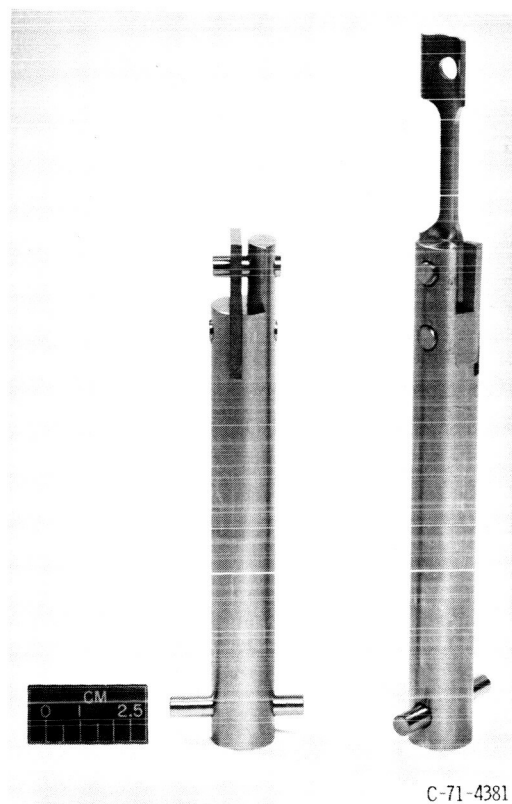


Figure 3. - Tubing section tensile-test specimen and grips used to test unlined and tungsten-lined T-111 tubing.

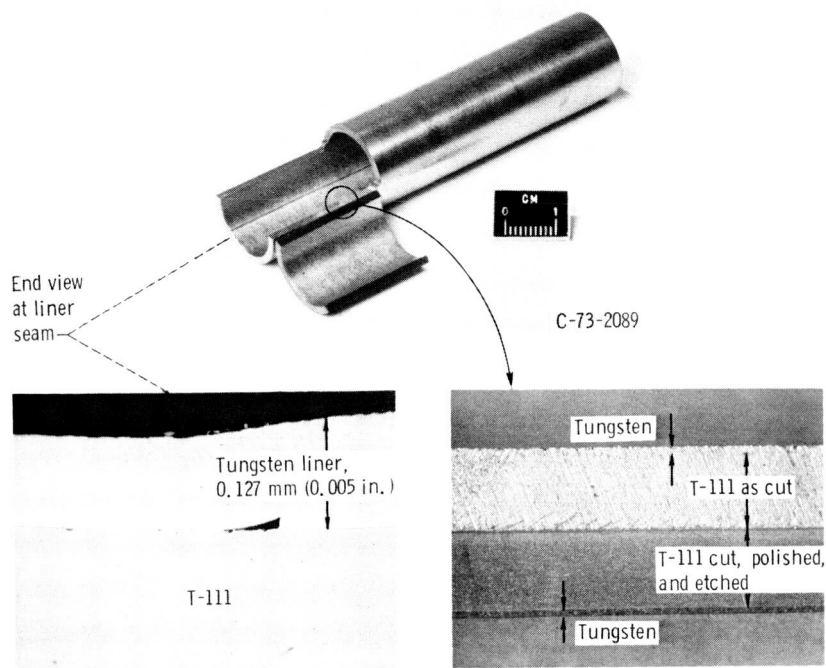
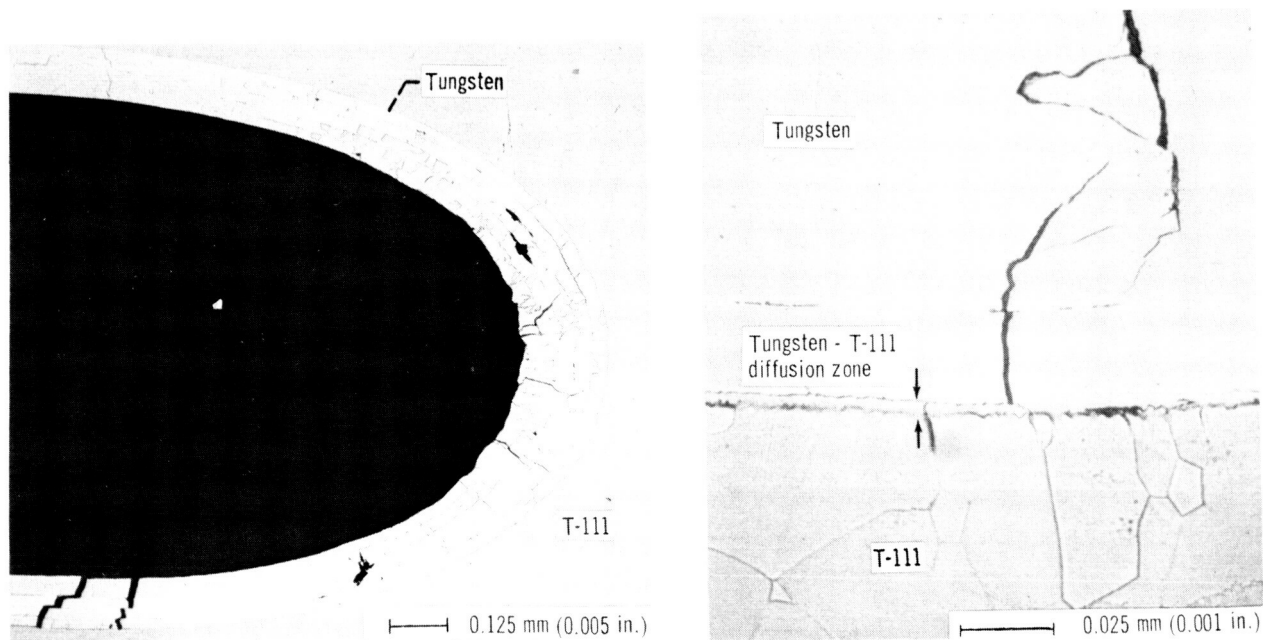


Figure 4. - Hot isostatically pressed tungsten-lined T-111 tubing, cut open with 120-grit silicon carbide cutoff wheel. (Inserts show cross-sectional view at tungsten seam and integrity after cutting.)



(a) Cross-sectional view of part of inner wall of lined tubing annealed in vacuum for 1 hour at 1315°C and flattened diametrically at room temperature. Unetched.

(b) Tubing annealed for 3 hours at 1650°C and flattened at room temperature. Etched to show tungsten - T-111 diffusion zone. Etchant: 50 cm³ nitric acid, 20 cm³ water, 30 g ammonium bifluoride.

Figure 5. - Photomicrographs of tungsten-lined T-111 tubing after annealing and flattening.

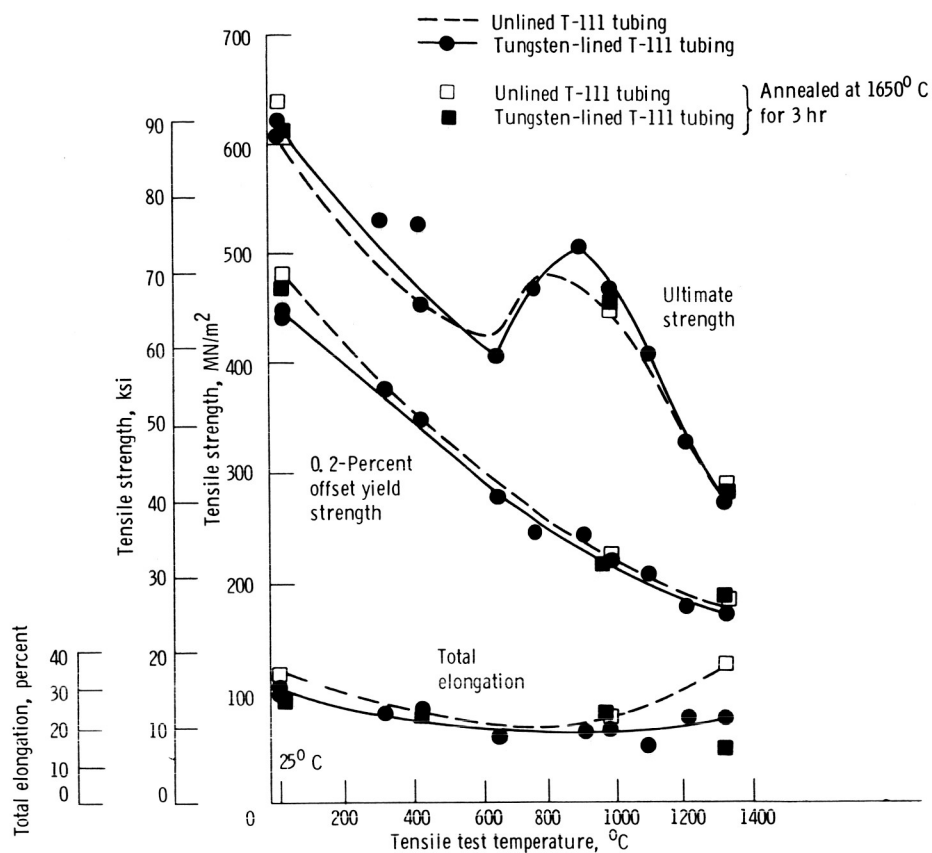


Figure 6. - Tensile properties of tungsten-lined and unlined T-111 tubing as a function of test temperature.

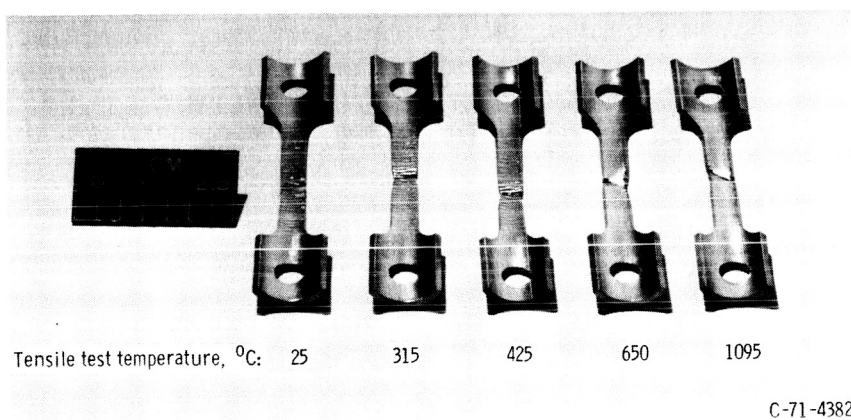
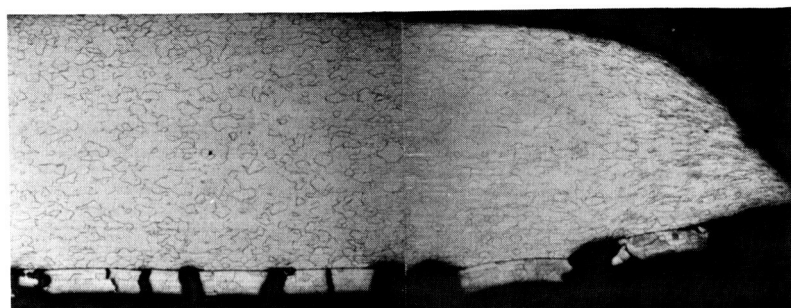
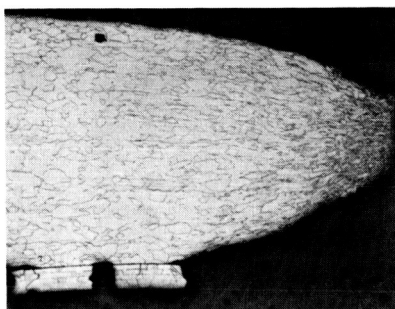


Figure 7. - Tungsten-lined T-111 tensile specimens after testing at various temperatures.

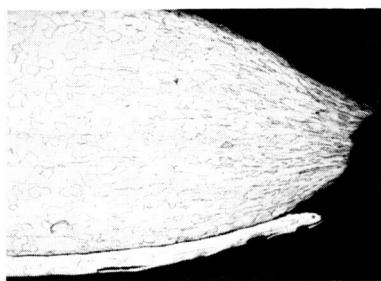


(a) 25° C.

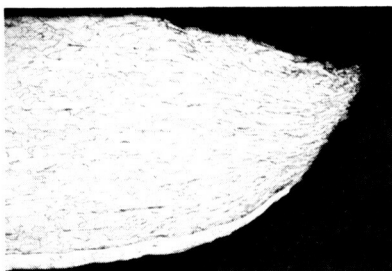
0.5 mm (0.02 in.)



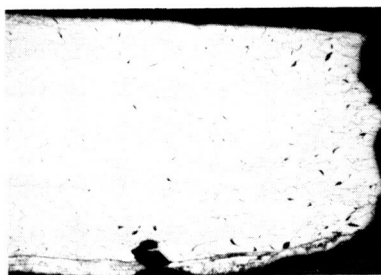
(b) 315° C.



(c) 650° C.



(d) 980° C.



(e) 1200° C.

Figure 8. - Fractured ends of tungsten-lined T-111 tubing tensile specimens after testing at indicated temperatures. Tungsten liner is at bottom in photomicrographs. Etchant for T-111: 50 cm³ nitric acid, 20 cm³ water, 30 g ammonium bifluoride. Etchant for tungsten: Murakami's reagent (10 g KOH, 10 g K₃FeCN₆, 100 cm³ water).